

PATENT ABSTRACTS OF JAPAN

S3

(11)Publication number : 11-288812

(43)Date of publication of application : 19.10.1999

(51)Int.Cl.

H01F 10/14

C04B 35/64

C23C 14/34

C23C 14/58

(21)Application number : 10-107100

(71)Applicant : SUMITOMO SPECIAL METALS CO LTD

(22)Date of filing : 01.04.1998

(72)Inventor : OKUDA TAKASHI
NAKANISHI AKIO
KIKUI FUMIAKI**(54) HIGH COERCIVE FORCE R-IRON-B THIN-FILM MAGNET AND MANUFACTURE THEREOF****(57)Abstract:**

PROBLEM TO BE SOLVED: To provide a high coercive force R-Fe-B thin-film magnet and a manufacturing method capable of substantially improving the coercive force in a film formation method by sputtering.

SOLUTION: By performing sputtering without heating substrate and performing the required crystallization heat treatment after forming an R-Fe-B alloy thin film on the substrate or further performing the required crystallization heat treatment after forming a protective film on the R-Fe-B alloy thin film, since a thin film will not be oxidized, this R-Fe-B thin-film magnet provided with a coercive force of larger than 10 kOe, larger than 25 kOe under preferred conditions, is obtained. By performing sputtering without heating the substrate, the thin-film magnet provided with the high coercive force can be obtained without the thin-film magnet becoming oxidized, the substrate modified or deformed, the structure of a device becoming complicated. Furthermore, by further forming a Ti protective film on the thin film formed on the substrate, oxidization at the heat treatment after the formation of the thin film is prevented, and the decline in the coercive force is prevented.

LEGAL STATUS

[Date of request for examination]

[Date of sending the examiner's decision of rejection]

[Kind of final disposal of application other than the examiner's decision of rejection or application converted registration]

[Date of final disposal for application]

[Patent number]

[Date of registration]

*** NOTICES ***

JPO and NCIP are not responsible for any damages caused by the use of this translation.

1. This document has been translated by computer. So the translation may not reflect the original precisely.
2. **** shows the word which can not be translated.
3. In the drawings, any words are not translated.

DETAILED DESCRIPTION

[Detailed Description of the Invention]

[0001]

[Field of the Invention] In case this invention relates to the thin film magnet used for a micro motor, a micro-actuator, a micro magnetometric sensor, etc., and the R-Fe-B system thin film magnet which has high coercive force especially and forms the R-Fe-B system (R is at least one sort of rare earth elements) alloy film on a substrate by sputtering, it relates to the high coercive force R-Fe-B system thin film magnet which acquired high coercive force, and its manufacture approach by forming membranes, without heating a substrate and preparing a protective coat further.

[0002]

[Description of the Prior Art] In recent years, thin shape-ization of the magnet used for a motor, an actuator, etc. is demanded with small and high-performance-izing of electronic equipment.

[0003] The R-Fe-B system permanent magnet is used abundantly at current and these applications. Although a R-Fe-B system magnet is the ingredient optimal from the outstanding magnetic properties for thin-shape-izing, a sintered magnet and a bond magnet is [the thickness of about hundreds of micrometers] limitations.

[0004] Then, research of thin-film-izing of a R-Fe-B system magnet is made, and it gets down, for example, Cadieu and others has reported in 1987 that coercive force obtained the thin film of 8-14kOe by RF spatter recently (6 Vac.Sci.Technol., A 1688 (1988)).

[0005] Moreover, Yamasaki and others has reported in 1990 that coercive force obtained the thin film of 3-7kOe by the DC magnetron sputtering method. Furthermore, Yamashita and others has got the thin film of coercive force 7kOe and residual magnetization 9.6kG by the DC magnetron sputtering method at the peak price in 1991 by the 65.5**77B10**Nd13**17Fe17.5 presentation (the Magnetics Society of Japan 15,241-244 (1991)).

[0006]

[Problem(s) to be Solved by the Invention] However, each above-mentioned approach has the problem that a substrate deteriorates or the structure of equipments, such as a problem of deforming, and temperature management, wiring of a substrate, becomes complicated, the problem that the soak nature of the substrate in heat treatment is still worse, etc. in order to heat a substrate.

[0007] Moreover, the coercive force of the obtained thin film is not yet practical. The highest in the inside of what is being cut by current is 14kOe(s). When thermal resistance etc. is taken into consideration since the magnet operating point is low to the degree of pole in case a R-Fe-B system thin film magnet is put in practical use, 14kOe(s) are insufficient for coercive force, and it is desirable. [of a more high thing]

[0008] This invention aims at offer of that manufacture approach in the high coercive force R-Fe-B system thin film magnet list which can raise coercive force remarkably in the membrane formation approach by sputtering for the purpose of offer of a R-Fe-B system thin film magnet with utilizable high coercive force.

[0009]

[Means for Solving the Problem] The result of wholeheartedly the research about the approach of not oxidizing the sputtering approach, especially the R-Fe-B system alloy thin film which forms membranes in order that artificers may attain the above-mentioned purpose, After performing sputtering, without heating a substrate and making a R-Fe-B system alloy thin film form on a

substrate The knowledge of the thin film magnet which there are no deterioration and deformation of a substrate by heat-treating on the optimal heat treatment conditions which can deposit a single domain particle, and has high coercive force being obtained from the amorphous thin film after necessary heat-of-crystallization processing, i.e., sputtering, was carried out.

[0010] Furthermore, in order to prevent the oxidation at the time of heat treatment after R-Fe-B system thin film membrane formation, by carrying out membrane formation of the protective coat further on a R-Fe-B system alloy thin film, artificers did the knowledge of fall prevention of coercive force being possible, and completed this invention.

[0011] That is, this invention is a high coercive force R-Fe-B system thin film magnet which has the R-Fe-B system (R is at least one sort of rare earth elements) alloy film which was formed in sputtering and heat-treated after membrane formation on the substrate, without heating a substrate, or is characterized by having a protective coat on this alloy film further.

[0012] Moreover, this invention is the manufacture approach of the high coercive force R-Fe-B system thin film magnet characterized by heat-treating, after performing sputtering and forming a R-Fe-B system (R is at least one sort of rare earth elements) alloy on a substrate, without heating a substrate, or after forming a protective coat on the alloy film further.

[0013]

[Embodiment of the Invention] In this invention, any well-known equipments, such as DC magnetron sputtering system usually used and RF sputtering system, can be used for sputtering. However, in this invention, since heating of a substrate is not needed, substrate heating apparatus etc. is not needed.

[0014] In this invention, as shown in the example mentioned later as target material for sputtering, the thing which dissolved and alloyed R, and Fe and B beforehand or the thing which has arranged each metal, for example, the thing which has arranged the chip of Nd and B on Fe plate, can be used.

[0015] What is necessary is just to determine the area which each metal in a target occupies so that it may be equivalent to the atomic ratio of the thin film magnet which it is going to obtain when arranging each metal which consists of R, Fe, and B and considering as a target. For example, to the area of the whole target, if it is the becoming presentation Nd30Fe54B16, Nd will arrange each metal so that Fe may occupy and B may occupy 16% of area 54% 30%.

[0016] Moreover, as a presentation of a thin film magnet, all of a well-known R-Fe-B system alloy presentation are employable as the presentation list of a target. what contains R 20at(s)% - 30at%, and contains 10at(s)% - 16at% for B in order to aim at high coercive force -- it is desirable, and since residual magnetization will fall if coercive force of R does not improve [less than / 20at% / and B] less than [10at%], but R exceeds and B exceeds 16at(s)% 30at(s)%, it is not desirable.

[0017] In this invention, any well-known substrate suitable for a magnetic application is employable as a substrate which forms a R-Fe-B system alloy thin film. By using Mo for some or all of a substrate for the purpose of antioxidizing of this alloy especially, emission of the oxygen from a substrate can be prevented, oxidation of a thin film can be controlled, and the fall of coercive force can be prevented.

[0018] As a concrete substrate, Mo plate, Mo radical alloy plate, the substrate that formed Mo or Mo radical alloy on various glass metallurgy group front faces can be mentioned. Moreover, as for the thickness of a substrate, it is desirable that it is 1mm or less.

[0019] As one of the descriptions of this invention, on the R-Fe-B system alloy thin film which formed membranes on the substrate by sputtering for the purpose of antioxidizing of a R-Fe-B system alloy thin film, as shown in an example, protective coats, such as Ti film, are formed.

[0020] That is, although it crystallizes by heat treatment since the R-Fe-B system alloy thin film after sputtering is in an amorphous condition, in the case of the heat-of-crystallization processing, R component in the oxygen in a heat treatment ambient atmosphere and a thin film etc. reacts, and there is a possibility that a thin film may oxidize. Therefore, by preparing a protective coat, oxidation of a thin film can be prevented and fall prevention of coercive force can be aimed at.

[0021] Being able to form Ti film shown in an example by the gaseous-phase forming-membranes methods, such as the sputtering method and vacuum deposition, as a protective coat, thickness is 100-1000A preferably several 10A - 100A of numbers.

[0022] It is the description that the manufacture approach of this invention does not heat a substrate during sputtering for oxidation control. As spread previously, all the thin film magnets proposed until now are performing sputtering, heating a substrate. Although it is an approach simple for this performing membrane formation and crystallization of a thin film at the time of **, and obtaining a thin film magnet, a substrate deteriorates, there is a problem of deforming, and the problem that the structure of equipment becomes complicated also has temperature management, wiring, etc. of a substrate. Furthermore, the problem of being bad also has the soak nature of the substrate in heat treatment.

[0023] Then, in this invention, after not heating a substrate but completing membrane formation of a thin film, it heat-treats anew in the case of sputtering, and it crystallizes a thin film. That is, since the thin film after sputtering is in an amorphous condition, it is necessary to heat-treat crystallization and the high magnetic properties which are the descriptions of a R-Fe-B system magnet can be made to discover by depositing an R2Fe14B ferromagnetism phase as a single domain particle from an amorphous phase.

[0024] Therefore, although heat treatment conditions can adopt the heat treatment conditions for the well-known recrystallization according to a presentation etc., as shown in an example, because of oxidation control, it is an ambient atmosphere in a vacuum and it is desirable in this invention, to carry out in temperature [of 600 degrees C - 650 degrees C] and time amount 30 minutes -, and 60 minutes.

[0025] Although there is especially no problem also in inert gas, in order to lessen oxygen as much as possible, once making a heat treatment ambient atmosphere into a vacua, it is desirable [an ambient atmosphere] to take the technique of carrying out Ar permutation. Moreover, heat treatment temperature does not have this better **, in order that lifting coercive force may decline the grain growth which crystallization is not enough and exceeds 650 degrees C at less than 600 degrees C. Although influenced by the amount of the class and gestalt of the furnace to process, and a heat-treated object etc., heat treatment time amount can almost be crystallized, if it is usually 30 minutes - about 60 minutes.

[0026] By the former, it becomes possible to obtain the R-Fe-B system thin film magnet which was not able to be obtained and which has high coercive force by using the manufacture approach by this invention mentioned above, an ingredient, etc. Specifically on 15 or more kOes and desirable conditions, the R-Fe-B system thin film magnet which has the coercive force of 25 or more kOes is obtained.

[0027]

[Example] Mo plate was used for the substrate formed [example 1] membranes, what has arranged the chip of Nd and B on Fe plate was used as the target, RF sputtering was performed on condition that ultimate-vacuum 2×10^{-6} Torr, Ar gas pressure 5×10^{-3} Torr, and high-frequency power 350W, without performing heating to a substrate, and the thin film magnet with which the thickness of about 1 micrometer and a presentation consist of Nd30Fe 54B16 (at%) was obtained.

[0028] Next, after forming Ti thin film of 300A thickness in sputtering on this thin film magnet, 650 degrees C and heat treatment for 30 minutes were performed in the vacuum.

[0029] The X diffraction result of the obtained thin film magnet is shown in drawing 1. It turns out that the diffraction peak of the Nd2Fe14 B phase of a ferromagnetic phase appears notably, and the R-Fe-B system thin film magnet is formed. Material identification was not made although phases other than Nd2Fe14 B phase were also accepted.

[0030] Moreover, the measurement result of magnetic properties is shown in drawing 2 at drawing 2. Drawing 2 is BH curve, an axis of ordinate shows residual magnetization and the axis of abscissa shows coercive force. With the pulse method, the test portion carried out 65kOe impression, magnetized to field inboard, and was measured using the oscillating sample mold magnetometer.

[0031] Magnetization was not set to 0, although the maximum magnetic field was impressed and measured to 16kOe(s) as shown in drawing 2. That is, the thin film magnet by this invention has the high coercive force of 16 or more kOes, and it is the value which was equivalent to the R-Fe-B system magnets (a sintered magnet, bond magnet, etc.) of bulk as for this, and was not acquired as a thin film magnet until now.

[0032] On the other hand, as for the R-Fe-B system thin film magnet by this invention, saturation

magnetization can expect improvement, when they reduce the volume ratio of the aforementioned unidentified phase by modification of sputtering conditions etc., although 3500G and residual magnetization were 3100G and were less than the property (14000G) of bulk.

[0033] The alloy which was beforehand ingoted to the substrate formed [example 2] membranes using what vapor-deposited Mo on quartz glass and with which a presentation consists of Nd30Fe 54B16 (at%) was made into target material, and RF sputtering was performed on the same conditions as an example 1. The thickness of the obtained thin film magnet was about 3 micrometers.

[0034] Subsequently, after forming Ti film of 500A thickness in sputtering on this thin film magnet, it heat-treated for 60 minutes at 600 degrees C among the vacuum. Coercive force was 25kOe(s) as a result of measuring using an oscillating sample mold magnetometer, after magnetizing the obtained thin film magnet like an example 1.

[0035] Mo plate was used for the substrate formed [example 3] membranes at the substrate, the alloy which was ingoted beforehand and with which a presentation consists of Nd20Fe 70B19 (at%) was made into target material, and RF sputtering was performed on the same conditions as an example 1. The thickness of the obtained thin film magnet was about 1 micrometer.

[0036] Subsequently, after forming Ti thin film of 200A thickness with vacuum deposition on this thin film magnet, it heat-treated for 30 minutes at 650 degrees C among the vacuum. Coercive force was 20kOe(s) as a result of measuring using an oscillating sample mold magnetometer, after magnetizing the obtained thin film magnet like an example 1.

[0037] As shown in the above example, the thin film magnet by this invention It has the high coercive force which was not able to be acquired until now, and sets to the presentation of a thin film magnet especially. The content of R 20at(s)% - 30at%, The content of B is 10at(s)% - 16at%, and when it has Ti thin film on a thin film magnet front face using the substrate which has Mo on Mo plate or a front face in a substrate, it becomes possible to acquire the outstanding coercive force of 15 or more kOes.

[0038]

[Effect of the Invention] The oxidation at the time of heat treatment after thin film membrane formation can prevent, and the fall of coercive force can prevent by obtaining the thin film magnet which has high coercive force, and forming Ti protective coat further on the thin film which formed membranes on the substrate, without oxidizing a thin film magnet by performing sputtering, without heating a substrate, a substrate's deteriorating and deforming, or the structure of equipment becoming complicated according to this invention.

[0039] Moreover, by using the substrate with which a part or all consists of Mo, emission of the oxygen from a substrate can be prevented, oxidation of a thin film can be controlled, and the fall of coercive force can be prevented. Furthermore, the R-Fe-B system thin film magnet which could deposit the R2Fe14B single domain particle efficiently, and was excellent in magnetic properties from the amorphous thin film after sputtering with the optimal heat treatment conditions can be obtained.

[0040] As mentioned above, the high coercive force R-Fe-B system thin film magnet by this invention is the optimal and indispensable to the application as which permanent magnets of a **** thin shape, such as a micro motor, a micro-actuator, and a micro magnetometric sensor, are required.

[Translation done.]

* NOTICES *

JPO and NCIPI are not responsible for any damages caused by the use of this translation.

1. This document has been translated by computer. So the translation may not reflect the original precisely.
2. **** shows the word which can not be translated.
3. In the drawings, any words are not translated.

TECHNICAL FIELD

[Field of the Invention] In case this invention relates to the thin film magnet used for a micro motor, a micro-actuator, a micro magnetometric sensor, etc., and the R-Fe-B system thin film magnet which has high coercive force especially and forms the R-Fe-B system (R is at least one sort of rare earth elements) alloy film on a substrate by sputtering, it relates to the high coercive force R-Fe-B system thin film magnet which acquired high coercive force, and its manufacture approach by forming membranes, without heating a substrate and preparing a protective coat further.

[Translation done.]

*** NOTICES ***

JPO and NCIPPI are not responsible for any damages caused by the use of this translation.

1. This document has been translated by computer. So the translation may not reflect the original precisely.

2. **** shows the word which can not be translated.

3. In the drawings, any words are not translated.

CLAIMS

[Claim(s)]

[Claim 1] The high coercive force R-Fe-B system thin film magnet which has the R-Fe-B system (R is at least one sort of rare earth elements) alloy film which was formed in sputtering and heat-treated after membrane formation on the substrate, without heating a substrate.

[Claim 2] The high coercive force R-Fe-B system thin film magnet which has a protective coat on a R-Fe-B system alloy film front face in claim 1.

[Claim 3] The manufacture approach of the high coercive force R-Fe-B system thin film magnet heat-treated after performing sputtering and forming a R-Fe-B system (R is at least one sort of rare earth elements) alloy on a substrate, without heating a substrate.

[Claim 4] The manufacture approach of the high coercive force R-Fe-B system thin film magnet heat-treated after forming a R-Fe-B system alloy and forming a protective coat further in claim 3.

[Translation done.]

(19)日本国特許庁 (JP)

(12) 公開特許公報 (A)

(11)特許出願公開番号

特開平11-288812

(43)公開日 平成11年(1999)10月19日

(51)Int.Cl.⁶H 01 F 10/14
C 04 B 35/64
C 23 C 14/34
14/58

識別記号

F I
H 01 F 10/14
C 23 C 14/34
14/58
C 04 B 35/64P
A
C

審査請求 未請求 請求項の数4 FD (全5頁)

(21)出願番号

特願平10-107100

(22)出願日

平成10年(1998)4月1日

特許法第30条第1項適用申請有り 1997年10月2日~10月5日 社団法人日本応用磁気学会主催の「第21回日本応用磁気学会学術講演会」において文書をもって発表

(71)出願人 000183417

住友特殊金属株式会社

大阪府大阪市中央区北浜4丁目7番19号

(72)発明者 奥田 高士

愛知県名古屋市北区名城二丁目2番城北住
宅2-41

(72)発明者 中西 昭男

大阪府三島郡島本町江川二丁目15-17 住
友特殊金属株式会社山崎製作所内

(72)発明者 菊井 文秋

大阪府三島郡島本町江川二丁目15-17 住
友特殊金属株式会社山崎製作所内

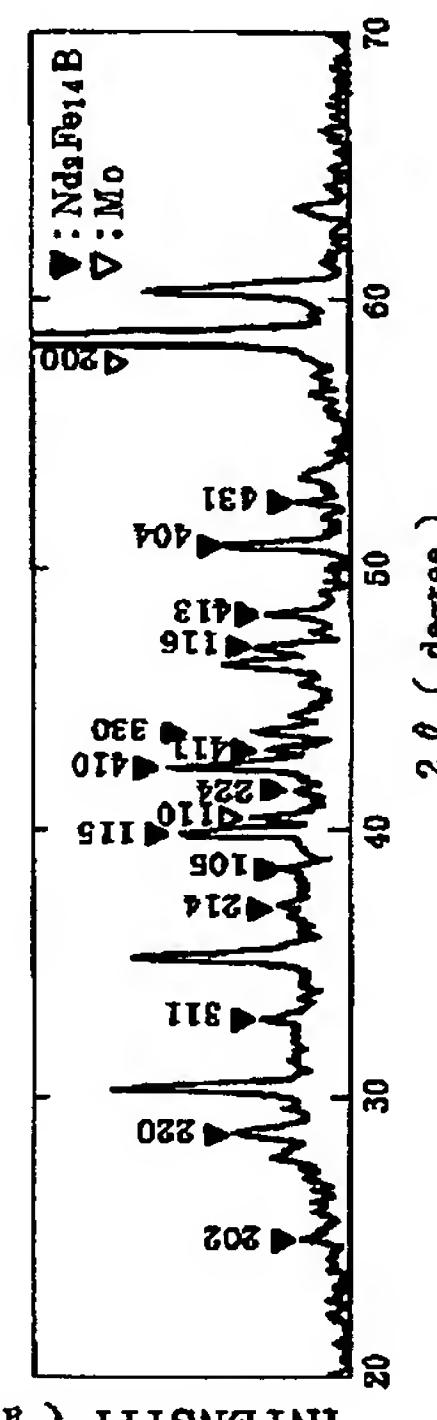
(74)代理人 弁理士 押田 良久

(54)【発明の名称】 高保磁力R-F e-B系薄膜磁石及びその製造方法

(57)【要約】

【課題】 スパッタリングによる成膜方法において、保磁力を著しく向上させることが可能な高保磁力R-F e-B系薄膜磁石並びにその製造方法の提供。

【解決手段】 基板を加熱せずにスパッタリングを行い、基板上にR-F e-B系合金薄膜を形成させた後に所要の結晶化熱処理するか、あるいはR-F e-B系合金薄膜上にさらに保護膜を成膜形成後に所要の結晶化熱処理することにより、薄膜を酸化させることができないため、15kOe以上、好ましい条件では25kOe以上の保磁力を有するR-F e-B系薄膜磁石が得られる。



【特許請求の範囲】

【請求項1】 基板上に、基板を加熱することなくスパッタリングにて成膜されかつ成膜後に熱処理されたR-F e-B系（Rは希土類元素の少なくとも1種）合金膜を有する高保磁力R-F e-B系薄膜磁石。

【請求項2】 請求項1において、R-F e-B系合金膜表面に保護膜を有する高保磁力R-F e-B系薄膜磁石。

【請求項3】 基板を加熱することなくスパッタリングを行い、基板上にR-F e-B系（Rは希土類元素の少なくとも1種）合金を成膜した後、熱処理する高保磁力R-F e-B系薄膜磁石の製造方法。

【請求項4】 請求項3において、R-F e-B系合金を成膜した後、さらに保護膜を成膜した後、熱処理する高保磁力R-F e-B系薄膜磁石の製造方法。

【発明の詳細な説明】

【0001】

【発明の属する技術分野】 この発明は、マイクロモータ、マイクロアクチュエータ、マイクロ磁気センサなどに用いられる薄膜磁石、特に、高保磁力を有するR-F e-B系薄膜磁石に係り、スパッタリングにより基板上にR-F e-B系（Rは希土類元素の少なくとも1種）合金膜を成膜する際に、基板を加熱することなく成膜し、さらに保護膜を設けることにより、高保磁力を得た高保磁力R-F e-B系薄膜磁石とその製造方法に関する。

【0002】

【従来の技術】 近年、電子機器の小型・高性能化に伴い、モータやアクチュエータなどに使用される磁石の薄型化が要求されている。

【0003】 現在、それら用途にはR-F e-B系永久磁石が多用されている。R-F e-B系磁石は、その優れた磁気特性から、薄型化には最適な材料ではあるが、焼結磁石、ボンド磁石共に数百 μm 程度の厚みが限界である。

【0004】 そこで、最近、R-F e-B系磁石の薄膜化の研究がなされおり、例えば、C a d i e uらは、1987年に、RFスパッタ法により、保磁力が8～14kOeの薄膜を得たことを報告している（V a c. S c i. T e c h n o l. , A 6, 1688 (1988)）。

【0005】 また、山崎らは、1990年に、DCマグネットロンスパッタ法により、保磁力が3～7kOeの薄膜を得たことを報告している。さらに、1991年には、山下らが、DCマグネットロンスパッタリング法により、Nd₁₃Fe_{65.5}B₁₀組成で、最高値で、保磁力7kOe、残留磁化9.6kGの薄膜を得ている（日本応用磁気学会誌 15, 241-244 (1991)）。

【0006】

【発明が解決しようとする課題】 しかし、上記の方法は、いずれも基板を加熱するため、基板が変質したり変形するなどの問題、また、基板の温度管理や配線など装置の構造が複雑になるという問題、さらに熱処理における基板の均熱性が悪いという問題などがある。

【0007】 また、得られた薄膜の保磁力がいまだ実用的ではない。現在分かっているものの中での最高が14kOeである。R-F e-B系薄膜磁石を実用化する際は、磁石動作点が極度に低いため、耐熱性などを考慮すると、保磁力は14kOeでは足りず、より高いことが望ましい。

【0008】 この発明は、実用化可能な高保磁力を有したR-F e-B系薄膜磁石の提供を目的とし、スパッタリングによる成膜方法において、保磁力を著しく向上させることが可能な高保磁力R-F e-B系薄膜磁石並びにその製造方法の提供を目的とする。

【0009】

【課題を解決するための手段】 発明者らは、上記の目的を達成するためスパッタリング方法、特に成膜するR-F e-B系合金薄膜を酸化させない方法について鋭意研究の結果、基板を加熱せずにスパッタリングを行い、基板上にR-F e-B系合金薄膜を形成させた後に、所要の結晶化熱処理、すなわちスパッタリング後のアモルファス薄膜から、単磁区粒子を析出させることができる最適な熱処理条件で熱処理することにより、基板の変質や変形がなく、かつ高い保磁力を有する薄膜磁石が得られることを知見した。

【0010】 さらに、発明者らは、R-F e-B系薄膜成膜後の熱処理時の酸化を防止するために、R-F e-B系合金薄膜上にさらに保護膜を成膜形成することにより、保磁力の低下防止が可能であることを知見し、この発明を完成した。

【0011】 すなわち、この発明は、基板上に、基板を加熱することなくスパッタリングにて成膜されかつ成膜後に熱処理されたR-F e-B系（Rは希土類元素の少なくとも1種）合金膜を有する、あるいはさらに該合金膜上に保護膜を有することを特徴とする高保磁力R-F e-B系薄膜磁石である。

【0012】 また、この発明は、基板を加熱することなくスパッタリングを行い、基板上にR-F e-B系（Rは希土類元素の少なくとも1種）合金を成膜した後、あるいはさらに合金膜上に保護膜を成膜した後、熱処理することを特徴とする高保磁力R-F e-B系薄膜磁石の製造方法である。

【0013】

【発明の実施の形態】 この発明において、スパッタリングには、通常用いられるDCマグネットロンスパッタリング装置、RFスパッタリング装置等、公知のいずれの装置も使用できる。但し、この発明においては、基板の加熱を必要としないため、基板加熱装置などは必要としな

い。

【0014】この発明において、スパッタリング用ターゲット材としては、後述する実施例に示す如く、予めRとFeとBを溶解し合金化したもの、あるいは個々の金属を配置したもの、例えば、Fe板上にNd及びBのチップを配置したものなどを用いることができる。

【0015】R、Fe、Bとからなる個々の金属を配置してターゲットとする場合、得ようとする薄膜磁石の原子比に相当するように、ターゲットにおける個々の金属が占める面積を決定すればよい。例えば、Nd₃₀Fe₅₄B₁₆なる組成であれば、ターゲット全体の面積に対して、Ndが30%、Feが54%、Bが16%の面積を占めるように各金属を配置する。

【0016】また、ターゲットの組成並びに薄膜磁石の組成としては、公知のR-Fe-B系合金組成のいずれをも採用できる。高保磁力を目的とするには、Rを20at%~30at%、Bを10at%~16at%を含有するもの好ましく、Rが20at%未満及びBが10at%未満では保磁力が向上せず、Rが30at%、Bが16at%を超えると残留磁化が低下するため好ましくない。

【0017】この発明において、R-Fe-B系合金薄膜を成膜する基板としては、磁石の用途に適した公知のいずれの基板も採用できる。特に該合金の酸化防止を目的に基板の一部あるいは全部にMoを用いることにより、基板からの酸素の放出が防止でき、薄膜の酸化を抑制して、保磁力の低下を防ぐことができる。

【0018】具体的な基板としては、Mo板、Mo基合金板、各種ガラスや金属表面にMoあるいはMo基合金を成膜した基板などを挙げることができる。また、基板の厚みは1mm以下であることが望ましい。

【0019】この発明の特徴の一つとして、R-Fe-B系合金薄膜の酸化防止を目的にスパッタリングにより基板上に成膜したR-Fe-B系合金薄膜上に、実施例に示す如く、Ti膜等の保護膜を形成する。

【0020】すなわち、スパッタリング後のR-Fe-B系合金薄膜はアモルファス状態であるため、熱処理によって結晶化するが、その結晶化熱処理の際に、熱処理雰囲気中の酸素と薄膜中のR成分などが反応し、薄膜が酸化する恐れがある。そのため、保護膜を設けることにより、薄膜の酸化を防止し、保磁力の低下防止を図ることができる。

【0021】保護膜として、例えば実施例に示すTi膜は、スパッタリング法や蒸着法などの気相成膜法により形成することができ、厚みは、数10Å~数100Å、好ましくは100~1000Åである。

【0022】この発明の製造方法は、酸化抑制のため、スパッタリング中に基板を加熱しないことが特徴である。先に述べたように、これまで提案されている薄膜磁石は、全て基板を加熱しながらスパッタリングを行なつ

ている。これは、薄膜の成膜と結晶化を同時に行なうものであって、薄膜磁石を得るには簡便な方法ではあるが、基板が変質したり変形するなどの問題があり、また、基板の温度管理や配線など、装置の構造が複雑になるという問題もある。さらに、熱処理における基板の均熱性が悪いという問題もある。

【0023】そこで、この発明においては、スパッタリングの際は基板を加熱せず、薄膜の成膜が終了した後、改めて熱処理を行ない、薄膜を結晶化させる。すなわち、スパッタリング後の薄膜はアモルファス状態であるため、結晶化の熱処理を施す必要があり、アモルファス相からR₂Fe₁₄B強磁性相を単磁区粒子として析出させることにより、R-Fe-B系磁石の特徴である高磁気特性を発現させることができる。

【0024】従って、この発明においては、熱処理条件は組成等に応じた公知の再結晶化のための熱処理条件が採用可能であるが、実施例に示す如く、酸化抑制のため、真空中雰囲気で、温度600℃~650℃、時間30分~60分で行なうことが好ましい。

【0025】熱処理雰囲気は、不活性ガス中でも特に問題はないが、酸素を極力少なくするために、一旦真空状態にした後、Ar置換する手法をとることが望ましい。また、熱処理温度は、600℃未満では結晶化が十分でなく、また650℃を超えると粒成長を起こし保磁力が低下するためこのましくない。熱処理時間は、処理する炉の種類や形態、被熱処理物の量などによって左右されるが、通常30分~60分程度であればほとんど結晶化することができる。

【0026】上述したこの発明による製造方法、材料などを用いることにより、従来では得ることができなかつた、高保磁力を有するR-Fe-B系薄膜磁石を得ることが可能となる。具体的には、15kOe以上、好ましい条件では25kOe以上の保磁力を有するR-Fe-B系薄膜磁石が得られる。

【0027】

【実施例】実施例1

被成膜基板にMo板を用いて、Fe板上にNd及びBのチップを配置したものをターゲットとし、到達真空度2×10⁻⁶Torr、Arガス圧5×10⁻³Torr、高周波電力350Wの条件で、基板への加熱を行わずにRFスパッタリングを行い、厚み約1μm、組成がNd₃₀Fe₅₄B₁₆(at%)からなる薄膜磁石を得た。

【0028】次に、該薄膜磁石上に、300Å厚みのTi薄膜をスパッタリングにて成膜した後、真空中で650℃、30分間の熱処理を施した。

【0029】得られた薄膜磁石のX線回折結果を図1に示す。強磁性相のNd₂Fe₁₄B相の回折ピークが顕著に現れ、R-Fe-B系薄膜磁石が形成されていることが分かる。Nd₂Fe₁₄B相以外の相も認められたが、物質の同定はできなかった。

【0030】また、図2に磁気特性の測定結果を図2に示す。図2はBHカーブであり、縦軸は残留磁化を、横軸は保磁力を示している。測定試料は、パルス法で面内方向に65kOe印加して着磁し、振動試料型磁力計を用いて測定した。

【0031】図2に示す如く、最大磁場を16kOeまで印加して測定したが、磁化は0にならなかった。すなわち、この発明による薄膜磁石は、16kOe以上の高保磁力を有しており、これは、バルク（焼結磁石、ボンド磁石等）のR-F e-B系磁石と同等であって、薄膜磁石としてはこれまで得られなかった値である。

【0032】一方、この発明によるR-F e-B系薄膜磁石は、飽和磁化は3500G、残留磁化は3100Gであり、バルクの特性（14000G）には及ばなかったが、スパッタリング条件などの変更によって、前記の未確認相の体積比を低減することにより、向上が見込まれる。

【0033】実施例2

被成膜基板に、石英ガラス上にMoを蒸着したものを用いて、予め溶製した、組成がNd₃₀Fe₅₄B₁₆（at%）からなる合金をターゲット材とし、実施例1と同じ条件でRFスパッタリングを行った。得られた薄膜磁石の厚みは約3μmであった。

【0034】次いで、該薄膜磁石上に500Å厚みのTi膜をスパッタリングにて成膜した後、真空中600°Cで60分間熱処理した。得られた薄膜磁石を実施例1と同様に着磁した後、振動試料型磁力計を用いて測定した結果、保磁力は25kOeであった。

【0035】実施例3

被成膜基板に基板にMo板を用いて、予め溶製した、組成がNd₂₀Fe₇₀B₁₀（at%）からなる合金をターゲット材とし、実施例1と同じ条件でRFスパッタリングを行った。得られた薄膜磁石の厚みは約1μmであった。

【0036】次いで、該薄膜磁石上に200Å厚みのTi薄膜を蒸着法にて形成した後、真空中650°Cで30*

10

20

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

30

【図2】

